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# Lithium [(R,S)-N,N'-Ethylenediaminedisuccinato]cobaltate(III) Trihydrate

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**Abstract.** $Li[Co(C_{10}H_{12}N_2O_8)].3H_2O$, $C_{10}H_{12}CoN_2O_8^-.Li^+.3H_2O$, $M_r = 408.2$, monoclinic, $P2_1/c$, $a = 9.624$ (10), $b = 12.788$ (6), $c = 12.049$ (6) Å, $\beta = 94.85$ (6)°, $U = 1477.6$ (1.8) Å$^3$, $Z = 4$, $D_m = 1.80$ (2), $D_x = 1.83$ Mg m$^{-3}$, Mo $K\alpha$ ($\lambda = 0.7107$ Å), $\mu = 1.28$ mm$^{-1}$. The final $R$ is 0.074 for 1689 independent observed reflections. The Co atoms of the $[Co_2\{(R,S)\text{-edds}\}_2]$ dimeric complex anion are bonded octahedrally to the two N atoms and one O atom from each of the four carboxylate arms of the two complexing species. The complex anions are held together by $LiO_4$ tetrahedra and hydrogen bonds.

**Introduction.** Preparation, chemistry and preliminary results on the crystal structure of $Li[Co\{(R,S)\text{-edds}\}].3H_2O$ were given by Pavelčík & Majer (1977). In this paper full crystallographic data are presented. The crystal used for the structure determination was of an approximate cubic form {100}, {011} with an edge dimension of 0.2 mm. The intensities of 3785 independent reflections (2.9° ≤ 2θ ≤ 55°) were collected on a Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation and the $\theta$–$2\theta$ technique at a scan rate varying from 4.88 to 29.3° min$^{-1}$ in $2\theta$. The background was measured at each end of the scan for one half of the reflection scan time. Two standards, monitored after every 94 reflections,

showed that no correction for instrumental instability or crystal decay was required. 1689 reflections with $I > 1.96\sigma(I)$ were considered as observed (only 45%). A value of $0.698\sigma(I)$ was assigned to the weak unobserved reflections with $I < 0.698\sigma(I)$. The intensities were corrected for Lorentz and polarization factors. No corrections for absorption or extinction were made. The structure was solved by three-dimensional Patterson and electron density Fourier syntheses. H atoms were found for the $[(R,S)\text{-edds}]^{4-}$ anion, but not for the water molecules. The structure was refined by block-diagonal least squares with anisotropic thermal parameters for the non-hydrogen and isotropic thermal parameters for the H atoms. The function $\sum w(|F_o| - |F_c|)^2$ was minimized; a weighting scheme $w^{-1} = \sigma^2(|F_o|) + (C|F_o|)^2$, where $\sigma(|F_o|)$ is derived from counting statistics and $C = 0.05$, was employed. $C$ was adjusted so that constant values of $\langle w(|F_o| - |F_c|)^2 \rangle$ were obtained in different $|F_o|$ intervals. The final residual $R$ ($= \sum |\Delta F|/\sum |F_o|$) was 0.074 for the observed reflections used in the refinement and 0.166 including the zero-weighted reflections. Corresponding weighted residuals $R_w$ [ $= (\sum w|\Delta F|^2/\sum w|F_o|^2)^{1/2}$] were 0.083 and 0.114. The maximum peak in the final difference synthesis was 0.38 e Å$^{-3}$. Scattering factors were taken from International Tables for X-ray Crystallography (1968). All crystallographic calculations were performed with the NRC program package (Ahmed, 1970) on a Siemens 4004/150 computer. Atomic coordinates for the non-hydrogen

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Table 1. *Final atomic coordinates* (×10⁴) *for the non-hydrogen atoms*

E.s.d.'s are given in parentheses.

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| C(1) | 1637 (10) | 1982 (8) | −1381 (7) |
| C(2) | 3002 (9) | 1390 (8) | −1215 (8) |
| C(3) | 4351 (10) | 2150 (7) | 513 (8) |
| C(4) | 3418 (10) | 3043 (7) | 807 (8) |
| C(5) | 5078 (9) | 1685 (8) | 1577 (8) |
| C(6) | 4165 (10) | 1216 (8) | 2432 (8) |
| C(7) | −515 (9) | 2133 (7) | −340 (7) |
| C(8) | −781 (9) | 1973 (7) | 870 (8) |
| C(9) | −1809 (10) | 1884 (7) | −1125 (8) |
| C(10) | −2404 (10) | 831 (8) | −872 (7) |
| O(1) | 2134 (6) | 2808 (5) | 874 (5) |
| O(2) | 3935 (7) | 3906 (5) | 980 (6) |
| O(3) | 2851 (6) | 1148 (5) | 2215 (5) |
| O(4) | 4732 (7) | 919 (6) | 3318 (6) |
| O(5) | 163 (6) | 1537 (5) | 1497 (5) |
| O(6) | −1910 (6) | 2244 (6) | 1180 (6) |
| O(7) | −1491 (6) | 108 (5) | −735 (5) |
| O(8) | −3671 (7) | 723 (5) | −863 (6) |
| N(1) | 732 (7) | 1534 (6) | −556 (6) |
| N(2) | 3455 (7) | 1306 (6) | 1 (6) |
| Co(1) | 1840 (1) | 1348 (1) | 806 (1) |
| W(1) | 7999 (8) | 657 (6) | 3003 (7) |
| W(2) | 6892 (8) | 4170 (7) | 440 (7) |
| W(3) | 717 (11) | 106 (11) | 3448 (11) |
| Li(1) | 6512 (19) | 364 (13) | 3898 (15) |

atoms are listed in Table 1,* bond distances and angles in Table 2, and selected torsion angles in Table 3.

**Discussion.** The Li⁺ ion is surrounded by four O atoms at the corners of an irregular tetrahedron, two belonging to water molecules and two to the carboxylic groups of the [(R,S)-edds]⁴⁻ molecule. O—Li—O angles

* Lists of structure factors, thermal parameters and H atom positional parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35327 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *and angles* (°) *in the complex anion and* LiO₄ *tetrahedron with e.s.d.'s in parentheses*

See Table 4 for the symmetry code.

| | | | | |
|---|---|---|---|---|
| Co—N(1) | 1·895 (7) | C(5)—C(6) | 1·532 (13) |
| Co—N(2) | 1·901 (7) | C(6)—O(3) | 1·272 (11) |
| Co—O(1) | 1·889 (6) | C(6)—O(4) | 1·218 (11) |
| Co—O(3) | 1·901 (6) | C(7)—C(8) | 1·515 (12) |
| Co—O(5) | 1·893 (6) | C(7)—C(9) | 1·532 (13) |
| Co—O(7ⁱ) | 1·893 (6) | C(8)—O(5) | 1·262 (11) |
| N(1)—C(1) | 1·492 (12) | C(8)—O(6) | 1·228 (11) |
| N(1)—C(7) | 1·465 (11) | C(9)—C(10) | 1·505 (13) |
| N(2)—C(2) | 1·497 (12) | C(10)—O(7) | 1·277 (11) |
| N(2)—C(3) | 1·483 (12) | C(10)—O(8) | 1·228 (11) |
| C(1)—C(2) | 1·515 (13) | Li—O(2ⁿⁱ) | 1·923 (18) |
| C(3)—C(4) | 1·512 (13) | Li—O(4) | 1·929 (19) |
| C(3)—C(5) | 1·528 (14) | Li—W(1) | 1·901 (20) |
| C(4)—O(1) | 1·281 (11) | Li—W(2ᵛ) | 1·956 (20) |
| C(4)—O(2) | 1·221 (11) | | |

| | | | |
|---|---|---|---|
| N(1)—Co—N(2) | 89·2 (3) | O(1)—C(4)—O(2) | 125·6 (9) |
| N(1)—Co—O(1) | 89·1 (3) | C(3)—C(5)—C(6) | 118·0 (8) |
| N(1)—Co—O(3) | 176·6 (3) | C(5)—C(6)—O(3) | 120·3 (8) |
| N(1)—Co—O(5) | 85·8 (3) | C(5)—C(6)—O(4) | 118·1 (8) |
| N(1)—Co—O(7ⁱ) | 90·0 (3) | O(3)—C(6)—O(4) | 121·6 (9) |
| N(2)—Co—O(1) | 85·6 (3) | N(1)—C(7)—C(8) | 107·9 (7) |
| N(2)—Co—O(3) | 94·2 (3) | N(1)—C(7)—C(9) | 114·6 (7) |
| N(2)—Co—O(5) | 172·8 (3) | C(8)—C(7)—C(9) | 111·8 (7) |
| N(2)—Co—O(7ⁱ) | 95·7 (3) | C(7)—C(8)—O(5) | 117·7 (8) |
| O(1)—Co—O(3) | 91·8 (3) | C(7)—C(8)—O(6) | 118·7 (8) |
| O(1)—Co—O(5) | 89·2 (3) | O(5)—C(8)—O(6) | 123·6 (8) |
| O(1)—Co—O(7ⁱ) | 178·4 (3) | C(7)—C(9)—C(10) | 111·4 (7) |
| O(3)—Co—O(5) | 90·9 (3) | C(9)—C(10)—O(7) | 113·9 (8) |
| O(3)—Co—O(7ⁱ) | 89·1 (3) | C(9)—C(10)—O(8) | 119·9 (8) |
| O(5)—Co—O(7ⁱ) | 89·5 (3) | O(7)—C(10)—O(8) | 126·2 (9) |
| Co—N(1)—C(1) | 107·9 (5) | Co—O(1)—C(4) | 111·8 (6) |
| Co—N(1)—C(7) | 108·7 (5) | Co—O(3)—C(6) | 126·9 (6) |
| C(1)—N(1)—C(7) | 116·7 (7) | Co—O(5)—C(8) | 113·0 (6) |
| Co—N(2)—C(2) | 108·3 (5) | Co—O(7ⁱ)—C(10ⁱ) | 126·1 (6) |
| Co—N(2)—C(3) | 103·8 (5) | C(4ᵛⁱ)—O(2ᵛⁱ)—Li | 143·1 (8) |
| C(2)—N(2)—C(3) | 117·8 (7) | C(6)—O(4)—Li | 138·6 (9) |
| N(1)—C(1)—C(2) | 105·6 (7) | O(2ᵛⁱ)—Li—O(4) | 100·8 (9) |
| N(2)—C(2)—C(1) | 110·0 (8) | O(2ᵛⁱ)—Li—W(1) | 114·7 (9) |
| N(2)—C(3)—C(4) | 108·1 (7) | O(2ᵛⁱ)—Li—W(2ᵛ) | 104·3 (9) |
| N(2)—C(3)—C(5) | 105·9 (7) | O(4)—Li—W(1) | 114·3 (9) |
| C(4)—C(3)—C(5) | 109·6 (8) | O(4)—Li—W(2ᵛ) | 108·7 (9) |
| C(3)—C(4)—O(1) | 115·6 (8) | W(1)—Li—W(2ᵛ) | 112·9 (9) |
| C(3)—C(4)—O(2) | 118·7 (8) | | |

Table 3. *Selected torsion angles* (°) *in the* [Co₂{(RS)-edds}₂]²⁻ *complex anion*

| | | | | | |
|---|---|---|---|---|---|
| Co—N(1)—C(7)—C(8) | −24·5 (8) | C(1)—C(2)—N(2)—Co | 27·5 (9) | Co—N(1)—C(7)—C(9) | −149·7 (10) |
| N(1)—C(7)—C(8)—O(5) | 11·2 (10) | C(2)—N(2)—Co—N(1) | −2·4 (8) | N(1)—C(7)—C(9)—C(10) | 72·6 (10) |
| C(7)—C(8)—O(5)—Co | 8·2 (10) | N(2)—Co—N(1)—C(1) | −23·2 (8) | C(7)—C(9)—C(10)—O(7) | −46·0 (10) |
| C(8)—O(5)—Co—N(1) | −18·7 (8) | C(1)—C(2)—N(2)—C(3) | −89·9 (9) | C(9)—C(10)—O(7)—Coⁱ | −161·8 (10) |
| O(5)—Co—N(1)—C(7) | 24·1 (8) | C(2)—N(2)—C(3)—C(4) | 83·2 (9) | C(10)—O(7)—Coⁱ—N(1ⁱ) | −131·4 (10) |
| Co—N(2)—C(3)—C(5) | 81·0 (7) | C(4)—C(3)—C(5)—C(6) | 60·9 (11) | C(7)—N(1)—C(1)—C(2) | 165·1 (7) |
| N(2)—C(3)—C(5)—C(6) | −55·6 (10) | Co—N(2)—C(3)—C(4) | −36·5 (8) | C(8)—C(7)—N(1)—C(1) | −146·8 (7) |
| C(3)—C(5)—C(6)—O(3) | 5·3 (13) | N(2)—C(3)—C(4)—O(1) | 19·2 (11) | C(9)—C(7)—N(1)—C(1) | 88·0 (9) |
| C(5)—C(6)—O(3)—Co | 11·8 (13) | C(3)—C(4)—O(1)—Co | 9·2 (10) | C(8)—C(7)—C(9)—C(10) | −50·6 (10) |
| C(6)—O(3)—Co—N(2) | 13·0 (10) | C(4)—O(1)—Co—N(2) | −26·1 (7) | O(5)—C(8)—C(7)—C(9) | 138·1 (8) |
| O(3)—Co—N(2)—C(3) | −57·1 (7) | O(1)—Co—N(2)—C(3) | 34·4 (7) | C(2)—N(2)—C(3)—C(5) | −159·3 (7) |
| Co—N(1)—C(1)—C(2) | 42·3 (8) | O(7ⁱ)—Co—N(1)—C(7) | 113·6 (10) | C(5)—C(3)—C(4)—O(1) | −95·8 (10) |
| N(1)—C(1)—C(2)—N(2) | −45·5 (10) | | | | |
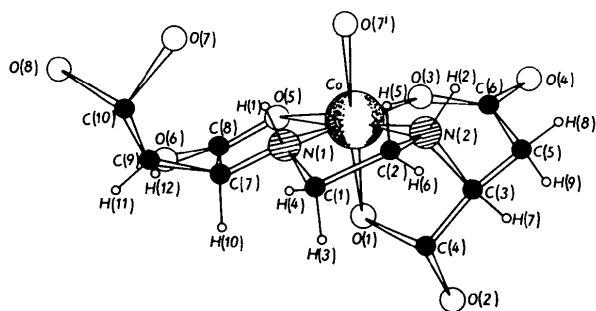
Fig. 1. A perspective drawing of the [Co{(R,S)-edds}]⁻ complex anion and the atom-numbering scheme.

Table 4. *Hydrogen bond contacts* (Å) ≤ 3 Å

| | | | |
|---|---|---|---|
| N(1)···O(7)* | 2·81 (1) | O(6)···$W$(1$^{ii}$) | 3·00 (1) |
| N(2)···O(8$^i$)* | 2·80 (1) | O(6)···$W$(2$^{ii}$) | 2·83 (1) |
| O(2)···$W$(2) | 2·99 (1) | $W$(1)···$W$(3$^{iii}$) | 2·72 (1) |
| O(3)···$W$(3) | 2·95 (1) | $W$(2)···$W$(3$^{iv}$) | 2·83 (2) |
| O(5)···$W$(3) | 2·99 (2) | | |

The equivalent positions are:

| | | | | |
|---|---|---|---|---|
| (i) | $-x,-y,-z$ | (iv) | $1-x, y+\frac{1}{2}, \frac{1}{2}-z$ |
| (ii) | $x-1, y, z$ | (v) | $x, \frac{1}{2}-y, \frac{1}{2}+z$ |
| (iii) | $x+1, y, z$ | (vi) | $1-x, y-\frac{1}{2}, \frac{1}{2}-z$. |

\* Intramolecular hydrogen bond.

range from 101 to 115° and Li—O distances are in the range 1·901–1·956 Å. The structure of the [Co{(R,S)-edds}]⁻ complex unit is shown in Fig. 1. The Co atom is bonded octahedrally to two N atoms and to four O atoms from four different carboxylate groups. Three O and two N donors are from one complexing molecule, the sixth O donor is from a centrosymmetrically related [Co{(R,S)-edds}]⁻ complex ion, forming in this way a dimeric unit. The stereochemistry of the [Co₂{(R,S)-edds}₂]²⁻ ion is very similar to that found in the crystal structure of Co[Co₂{(R,S)-edds}₂].14H₂O (Pavelčik, Soldánová & Majer, 1980). The geometries of the individual chelate rings can be deduced from Table 3. The glycine rings have asymmetric envelope conformations; the six-membered β-alanine chelate ring has a deformed half-chair conformation. The ethylenediamine ring is in an envelope conformation with C(1) out of the plane formed by the remaining four atoms [deviations of C(1) and C(2) from the N(1), Co, N(2) plane are −0·56 and 0·06 Å respectively]. The interatomic distances and angles listed in Table 2 are normal. Binuclear units transformed by the 2₁

symmetry operation are interconnected by LiO₄ tetrahedra which form a zigzag arrangement around the screw axis. The binuclear unit forms an analogous arrangement around another screw axis shifted by the translation (1,0,½) so that infinite layers parallel to the (102) plane are formed. The layers are connected by hydrogen bonds between H₂O molecules bonded to Li⁺ ions and uncoordinated O atoms of the carboxylic groups. The system of hydrogen bonds is described in Table 4. .

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